Short-Circuit Formation During NiCd Cell Reversal

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The reduction of $Cd(OH)_2$ to cadmium at the nickel electrode in the NiCd cell has been found to compete with the hydrogen evolution reaction during cell reversal. This reduction reaction can lead to internal short circuits as cadmium dendrites bridge the separator. The cadmium metal short circuits are reoxidized when the cell is recharged and do not appear to have a significant short-term effect on cell performance.

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## ACKNOWLEDGMENTS

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FIGURES

I. INTRODUCTION

The reversal of individual NiCd cells during battery operation is a situation that has long been recognized as being potentially dangerous to the successful operation of these batteries. During reversal of positive limited NiCd cells, hydrogen gas is typically evolved at the nickel electrode. If the NiCd cell is sealed, hydrogen pressure can build up to a point where the cell may fail. Cell reversal has been a particular concern in satellite NiCd battery operation, and consequently satellite power control systems have normally been designed to preclude the possibility of cell reversal. However, the use of deep discharge battery reconditioning (battery discharge to less than 0.1 V/cell) for currently proposed satellites raises concerns about long-term, low-rate cell reversal. During deep discharge reconditioning, imperfect matching of cell capacities will result in the cells that have the lowest capacity being reversed at a low rate, with the extent of reversal being directly proportional to the imbalance or dispersion in cell capacities. In addition, NiCd cell reversal is possible at high rates if one cell in a battery has experienced degradation so severe that its capacity is prematurely depleted while the battery is supporting satellite loads. This risk is compounded by any unusual stress on the battery and increases with battery age and battery depth of discharge. Again, proposed satellites having long-term missions with the batteries operating up to 80% depth of discharge are particularly susceptible to cell depletion and reversal at high rates while the battery is supporting satellite loads.

The ability to evaluate the risks associated with cell reversal is therefore essential to a broad spectrum of Space Division satellite programs. Proper risk evaluation requires a thorough understanding of the chemical and physical changes that take place in the NiCd cell as a result of reversal, as well as how these changes affect cell and battery performance. In this report the results of laboratory experiments are presented that help clarify the changes that are brought about inside the NiCd cell by reversal. The differences in NiCd cell behavior for low-current and high-current reversals will

be specifically examined in terms of the internal cell chemistry. For the application of these results to satellite battery situations, the implications insofar as limiting internal hydrogen evolution will be detailed. These results may provide some indication as to how repeated cell reversals affect performance, especially over the short term. However, a realistic indication of the effects of reversal on long-term battery performance can only come from appropriately designed battery life tests.

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II. BACKGROUND

The NiCd cell is designed so that the cell capacity is normally limited by depletion of the nickel electrode. Therefore, during both discharge and reversal, the reaction at the cadmium electrode is normally oxidation of cadmium metal to $Cd(OH)_2$

$$Cd + 2OH^- \xrightleftharpoons[\text{charge}]{\text{discharge}} Cd(OH)_2 + 2e^- \qquad E_o = 0.809 \qquad (1)$$
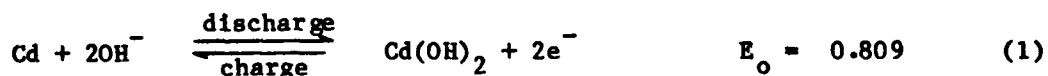
If the supply of active cadmium metal is depleted, the potential of the cadmium electrode drops to allow the oxygen evolution reaction to occur

$$4OH^- + 2H_2O + O_2 + 4e^- \qquad E_o = -0.436 \qquad (2)$$

Because of the normal large excess of cadmium over the nickel electrode capacity, reaction (2) should not occur during cell reversal unless the cell is vented or passivation of the cadmium electrode occurs.

During discharge of the nickel electrode, reduction of NiOOH occurs

$$NiOOH + H_2O + e^- \xrightleftharpoons[\text{charge}]{\text{discharge}} Ni(OH)_2 + OH^- \qquad E_o = 0.48 \qquad (3)$$
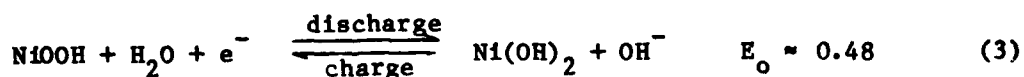
Reversal takes place upon depletion of the active NiOOH. The point at which reversal actually occurs is quite sensitive to discharge current, since much more of the NiOOH is actively available at lower discharge rates than at higher discharge rates. Complete discharge of the NiOOH semiconductor material to the divalent state does not actually occur because $Ni(OH)_2$ is an electronic insulator. For this reason it is expected that the reactions that occur during reversal and their rates will depend on the extent of depletion of NiOOH, and thereby the conductivity of the nickel electrode active material.

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The reactions that may occur at the nickel electrode during reversal are documented in the battery literature, and a brief review of previous work in this area is helpful. Hydrogen evolution at the nickel electrode has been widely studied, most recently by Ritterman[1] and by Badcock and Martinelli.[2]

$$2H_2O + 2e^- \xrightleftharpoons{\hspace{1cm}} 2OH^- + H_2 \qquad\qquad E_o = + 0.793 \qquad (4)$$

This reaction may occur at quite high rates on the discharged nickel electrode, and therefore is likely to involve the surfaces of the nickel sinter rather than the discharged nickel active material. Recombination of hydrogen in the NiCd cell can occur chemically by a mechanism such as that proposed by Ritterman[1]. Ritterman also claims to have observed a very rapid electrochemical hydrogen recombination reaction[3]. However, no evidence for this reaction has been observed by any other workers, and its mechanism has not been experimentally characterized by Ritterman.[1] Extensive studies of the rates of hydrogen recombination in the NiCd cell by Badcock and Martinelli[2] indicate that this reaction fits the first order catalytic mechanism proposed by Ritterman[1]. They obtained reaction rates and an activation energy for the recombination process. The rate of this reaction is quite slow, having a half-life of several days at 20°C.

It is commonly observed that, prior to hydrogen evolution, an induction period may take place after driving a NiCd cell into reversal and that hydrogen evolution, after it reaches a steady state, often does not proceed with 100% coulombic efficiency. Badcock and Martinelli[2] suggest that discharge of residual active material at high overpotentials can lead to the induction period. The lack of 100% coulombic efficiency for steady-state hydrogen evolution suggests that an additional reaction may be competing with hydrogen evolution during reversal. Rampel[*] has suggested that reduction of $Cd(OH)_2$ at the nickel electrode can occur during cell reversal by the reverse of reaction

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[*]G. Rampel, private communication.

(1). The $Cd(OH)_2$ is sometimes added to the nickel electrode during manufacturing (antipolar mass), but if not, is rapidly acquired from the cadmium electrode by migration of the somewhat soluble $Cd(OH)_2$.[4] Reduction of $Cd(OH)_2$ may therefore compete with hydrogen evolution under some conditions. Badcock and Martinelli[2] have noted that long-term cell reversal can result in the sudden development of what they claim to be a short-circuit condition. Rampel has suggested that this behavior results from a short circuit formed by the growth of cadmium metal dendrites from the nickel electrode to the cadmium electrode by reaction (5). Similar results have been observed by Ritterman;[3] however, he ascribes these results to a rapid electrochemical reaction by which hydrogen recombines in the NiCd cell. The resolution of the controversy concerning the mechanisms for this reaction that appears to compete with hydrogen evolution is of primary interest in the experiments reported here.

III. EXPERIMENTAL

The two aerospace NiCd cells used in the experiments reported here were 10-Ah prismatic cells manufactured by General Electric Company. They had polypropylene separators, and cadmium was added to the nickel electrode as an antipolar mass. They were about 7 years old and had not been subjected to extensive cycling. The cells had been subjected to a number of reversals during the work of Badcock and Martinelli;[2] however, they did not suffer any changes in performance characteristics as a result of the repeated reversals. Each cell was fitted with a Setra Systems 0 to 500 psia pressure transducer. The transducers were attached to the negative electrode post, through which a 1/16-in. hole had been drilled. The experiments were performed with the cells at ambient temperature, $23 \pm 3°C$. Both a PAR 173D and a McKee-Pedersen MP-1024 potentiostat were used in the charge, discharge, and reversal of the cells.

Laboratory cells were constructed from two sintered cadmium plates and one sintered nickel plate from a 10-Ah General Electric NiCd cell. The nickel plate was wrapped in a separator, either Viscon or Pellon 2505, and then sandwiched between the two cadmium plates. This plate pack was placed in a Plexiglas container that was closed (not sealed) and purged with a slow flow of nitrogen. The reference electrode consisted of an Ag_2O-coated silver wire. The plate pack was compressed by Teflon sheets inserted into the holder. The electrolyte consisted of 31% KOH. This provided a NiCd cell having about 10% of the capacity of the 10-Ah aerospace cells and possessing very similar charge and discharge voltage characteristics. All chemical analyses of plate and separator components were performed using atomic absorption spectroscopy.

IV. CHARACTERIZATION OF REVERSAL PROCESSES

The discharge of a positive limited NiCd cell typically leaves appreciable amounts of residual capacity that continue to discharge as the cell enters reversal. Discharge of residual capacity can account for the induction periods that are often evident between the beginning of reversal and the start of hydrogen evolution. The cyclic voltammogram (Fig. 1) represents the processes that occur during nickel cell reversal in the absence of appreciable residual charge at the nickel electrode. Before the cyclic sweep, the cell was held in reversal at -0.28 V to allow residual capacity to be discharged. After about 45 min of reversal at this potential, 35 psi of hydrogen had been generated. The cell potential was then linearly increased to 0.9 V then linearly decreased to -0.30 V, (see the results in Fig. 1). The sweep rate was 0.25 mV/sec for both the positive and the negative direction. No hydrogen was initially evolved as the cell potential was swept into reversal from positive voltages; however, a large reversal current was observed. The large peaks about 0 V in Fig. 1 suggest that an electrochemical reaction with some degree of reversibility occurs at a potential of about 0 V for the NiCd cell. This process must occur in competition with hydrogen evolution during reversal.

To determine the chemical reaction involved in this process that competes with hydrogen evolution, the NiCd cell potential was held at -0.15 V, and the reversal current was monitored as a function of time. The current at this potential was attributed solely to reactions other than hydrogen evolution, since no pressure rise was observed under conditions where we have found the hydrogen recombination rate to be very slow. The reversal current as a function of time is shown in Fig. 2. The reversal current remains quite low for a long period of time, then it begins to rise, exhibiting significant electrochemical noise as it rises. Eventually the current reached 1 A (C/10 rate), after which the cell was reversed at a 1 A constant current for about 100 hr, as shown in Fig. 3. During this period of reversal, the cell voltage increased to about -20 mV. After this period of reversal, the cell was

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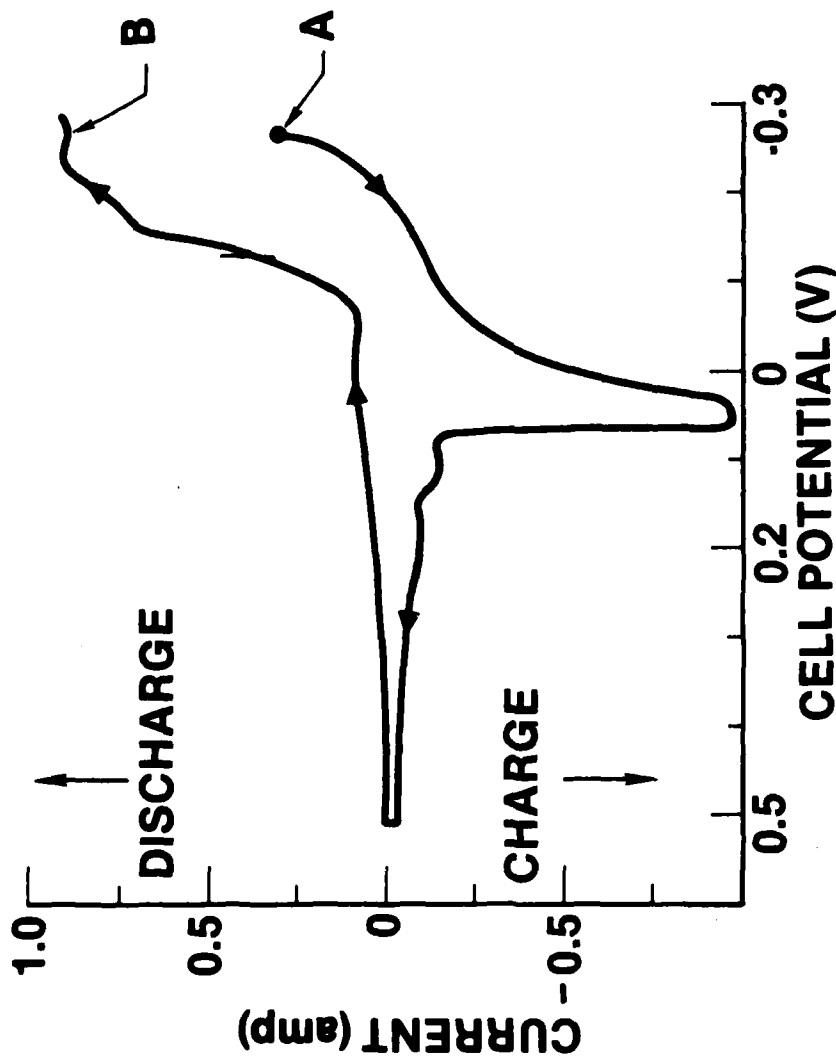
Fig. 1. Cyclic Voltammogram for NiCd Cell. Before sweep the cell was held at point A for 45 min, during which 35 psi of H_2 was generated. On the return sweep the first sign of H_2 generation was at point B.
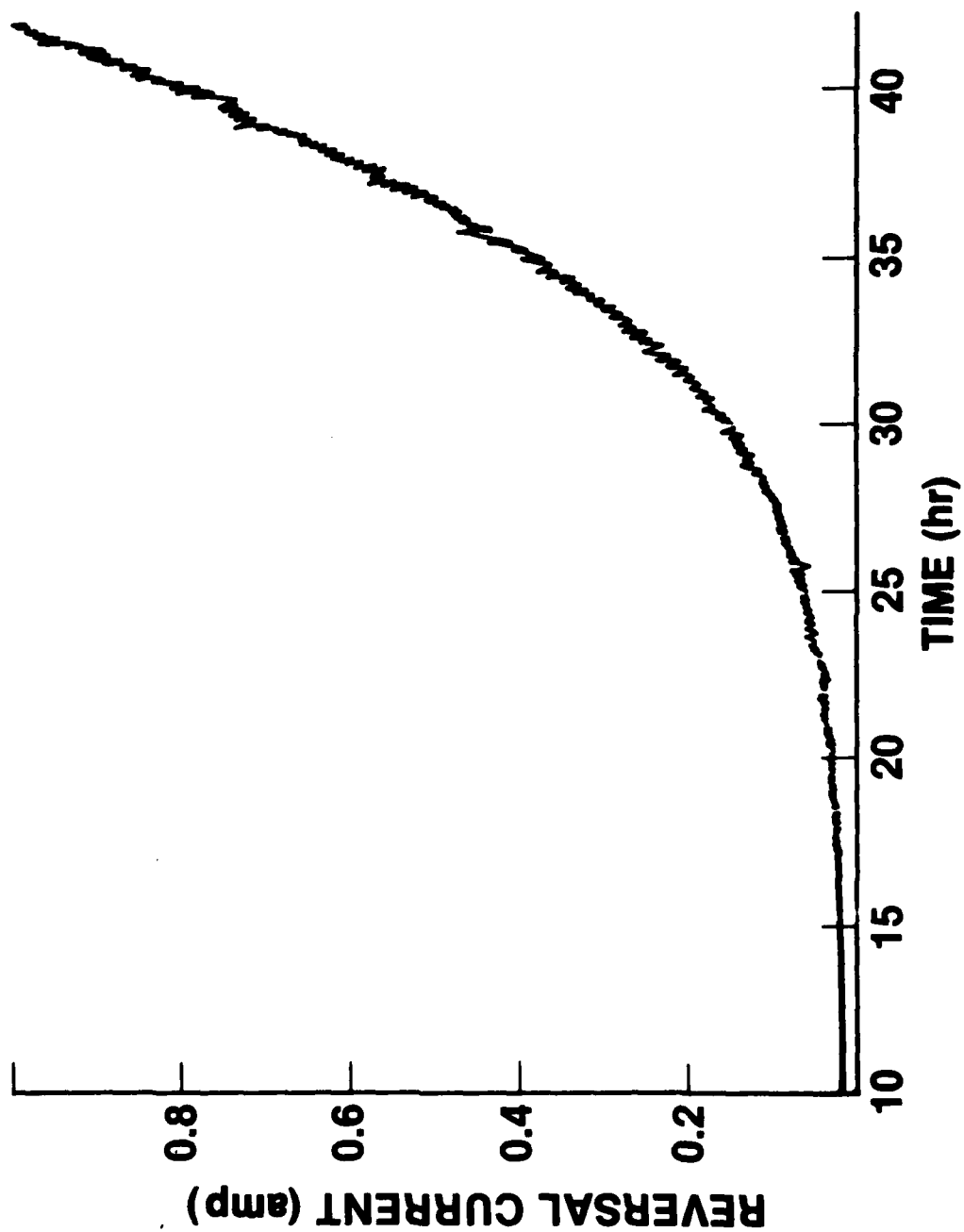
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Fig. 2. NiCd Cell Reversal Current as a Function of Time at Constant Cell Potential of -0.15 V.
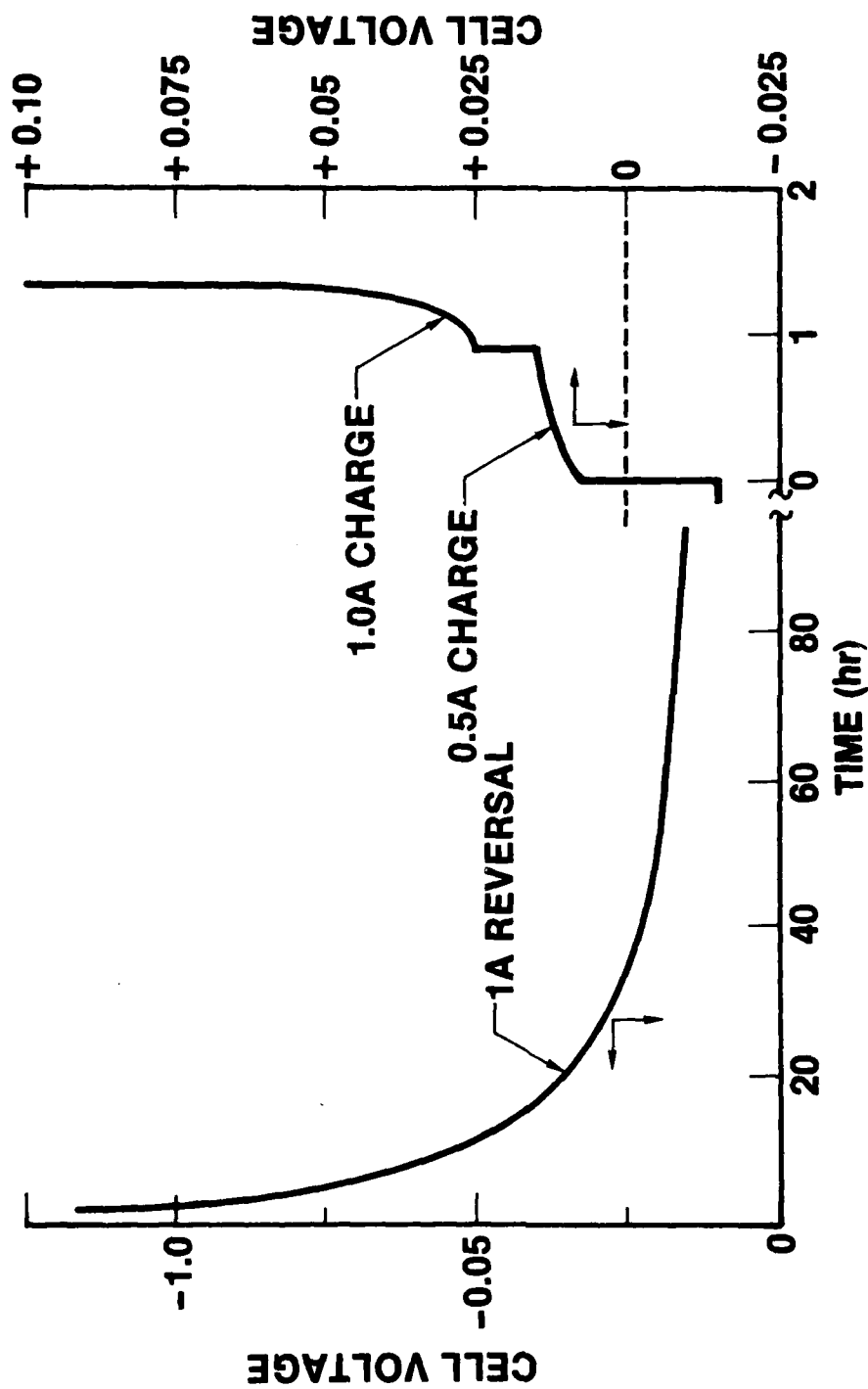
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Fig. 3. NiCd Cell Potential During 1-A Reversal for 100 hr, Followed by Recharge
for About 1.5 hr at 0.5 A, Then at 1.0 A.

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recharged (as indicated in Fig. 3), first at 0.5 A, then at 1.0 A. After more than 1 hr of recharge, during which the cell voltage remained below 30 mV, the voltage suddenly rose to 1.35 V, and the cell began to accept charge in an apparently normal fashion.

The behavior during cell reversal that is described above and indicated in Figs. 1 through 3 cannot be accounted for by residual capacity, since more than 100 Ah of charge were passed through the cell in reversal. No hydrogen evolution was observed at a 0.1 psia sensitivity. Another reaction that could take place during reversal at the nickel electrode is the reduction of the $Cd(OH)_2$ in the vicinity of the nickel electrode to cadmium metal. As this reaction would proceed at a low rate, dendritic crystallites of cadmium metal could grow from the conductive surfaces of the nickel electrode toward the cadmium electrode. When these growths make electrical contact with the cadmium electrode, a short circuit results, causing an increase in current in the constant potential experiment of Fig. 2. Equivalently a voltage drop occurs (Fig. 3) under conditions of constant current as this short circuit grows (resistance decreases). When recharge of the shorted cell is attempted, the cadmium crystallites should begin to oxidize. However this would not be an efficient process at low recharge currents since the short circuit carries most of the charging current and thereby maintains a low cell potential. Eventually the cadmium dendrites are oxidized through at their weakest points, and the short circuit is broken, permitting the cell to recharge.

Although the mechanism postulated here is consistent with the cell behavior during reversal, additional experiments were performed to verify these processes. These experiments included electrochemical impedance measurements during cell reversal and chemical analyses of the interelectrode areas following extensive cell reversal. A cell was constructe ' from one nicke ' and two cadmium plates from a 10-Ah NiCd cell and was reversed at the C/200 rate until the voltage suddenly rose above -0.10 V (about 16 hr were required). Subsequently the reversal current was increased in steps to the C rate over several days, never permitting the cell potential to drop below -0.05 V. No gas generation was observed during this sequence. After 1 day of reversal at the C rate, the cell was disassembled. The separators were found to contain heavy

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crystalline deposits of a dark material that contained cadmium as the sole metallic element. The deposits appeared to be heaviest adjacent to the nickel electrode. After exposure to air the black crystalline masses slowly (over several days) became a gray white flocculent material. Identical results were obtained using two different kinds of separator material, Pellon 2505 and Viscon. These materials were clean and unused before cell assembly. Both materials exhibited crystalline deposits in the separator, and in both cases almost identical cell behavior was observed during reversal.

V. IMPEDANCE OF REVERSED CELLS

The impedance of a 10-Ah cell was measured to determine that the impedance changes that occurred during reversal were consistent with the formation of an internal short circuit. These impedance measurements were made between 0 and 100 Hz using a galvanostatic technique that has been previously described.[5,6] Typical changes that take place in the cell impedance during reversal are indicated in Fig. 4(a). These measurements were performed 3, 20, and 54 hr into reversal at the C/50 rate; shorting, as noted by a sudden rise in cell voltage, was observed after about 6 hr of reversal. Therefore the impedance at 3 hr is for a cell having a negligible internal short circuit. After 6 hr, when the voltage began to suddenly increase, the impedance began to decrease, as indicated in Fig. 4(a). The lower frequency relaxation process in the impedance plot showed the most pronounced decrease during the reversal period.

To evaluate whether these changes in impedance are consistent with the formation of a short circuit, the impedance at 3 hr was also measured with external short circuits of 0.3 and 0.03 Ω. These results are shown in Fig. 4(b). The trend in the magnitude and frequency dispersion of the impedance is very similar to that in Fig. 4(a). Short-circuit formation during reversal is therefore consistent with the changes in impedance and potential that occur. Note that a quantitative comparison of Figs. 4(a) and 4(b) is not possible because data in Fig. 4(a) were obtained over several days, during which time the electrode impedances could have changed. The data in Fig. 4(b) on the other hand were all performed 3 hr into reversal, and therefore the internal cell impedance did not change significantly between these experiments.

An equivalent circuit for the NiCd cell during reversal is indicated in Fig. 5, where C_1, Z_{F1} and C_2, Z_{F2} are the double layer capacitance and the faradaic impedance of the positive and the negative electrodes, respectively; R_s the electrolyte resistance; and R_{sh} the resistance of a short-circuit path between the cell electrodes. In principle it should be possible to fit the
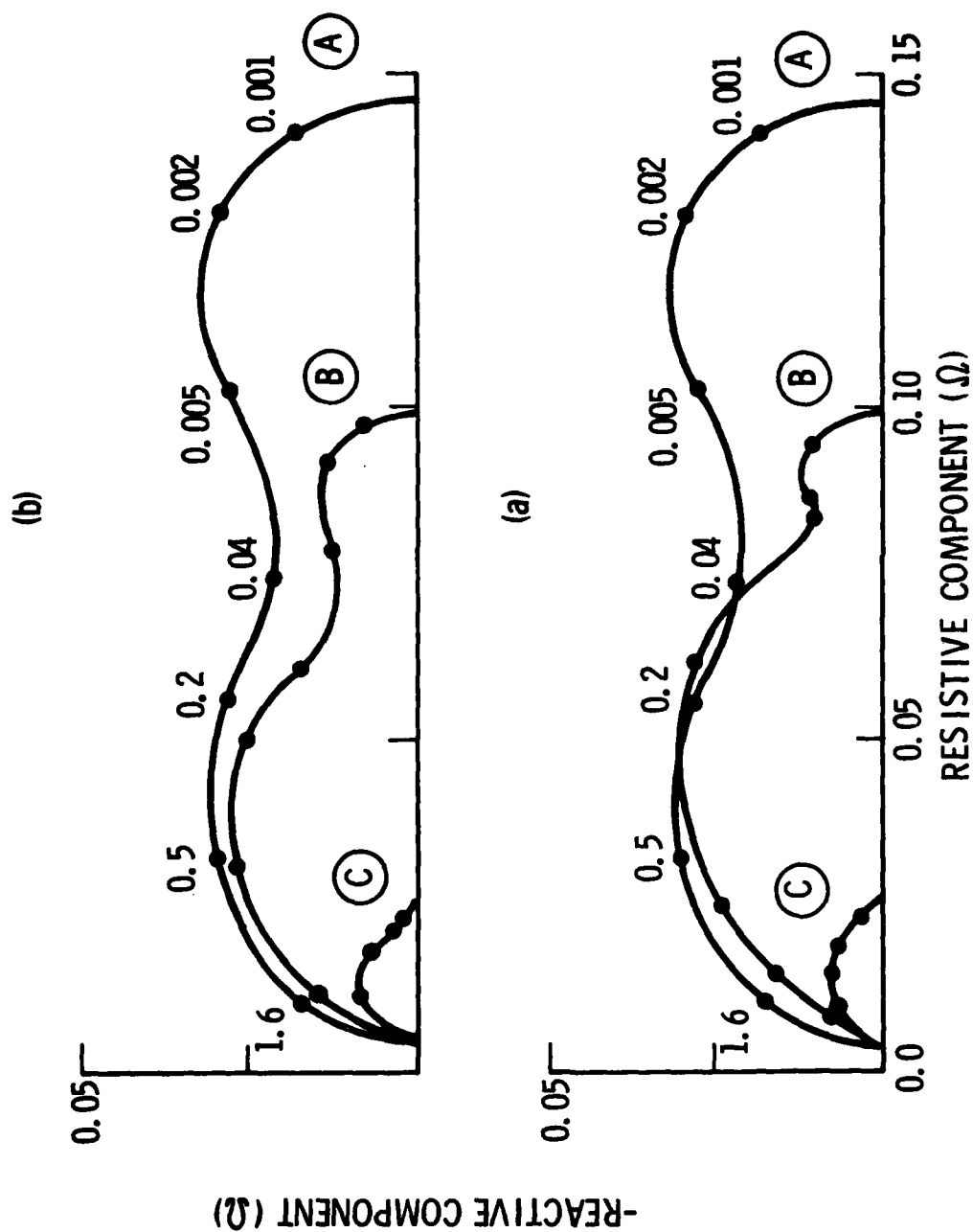
Fig. 4. NiCd Cell Impedance. (a) During reversal at 0.2 A (C/50). The numbers indicate frequency in Hz. Curve A is the impedance 3 hr into reversal, curve B 20 hr into reversal, and curve C 54 hr into reversal. (b) After 3 hr of reversal at 0.2 A (curve A), with external 0.3 ohm short circuit (curve B), and with external 0.03 ohm short circuit (curve C). The numbers indicate frequency in Hz.

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Fig. 5. Equivalent Circuit for NiCd Cell with
an Internal Short Circuit, R_{sh}.

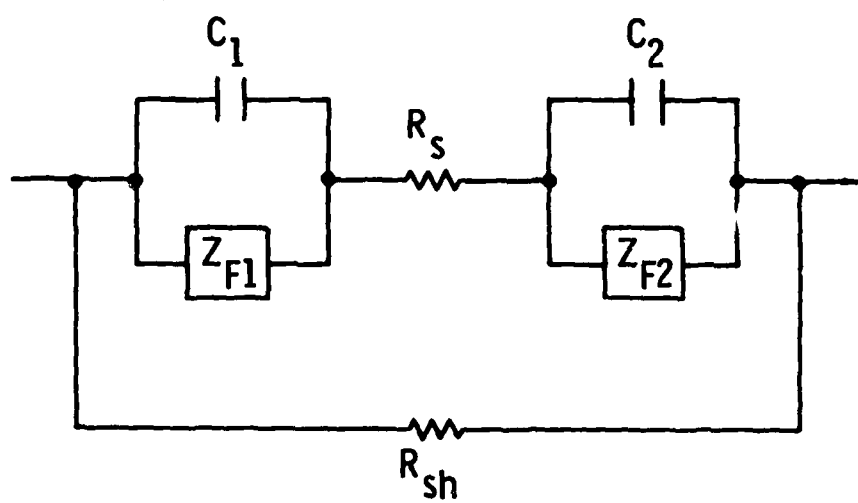impedance data to this equivalent circuit to determine the value of the short-circuit resistance R_{sh} at any time. However for this procedure to be practical, the faradaic impedances Z_{F1} and Z_{F2} must be accurately modeled. We found this to be extremely difficult because of the complexity of the system. Both the nickel and the cadmium electrode impedances include the effects of charge transfer and diffusion processes. In addition, before short-circuit formation it is likely that both hydrogen evolution and cadmium reduction reactions occur in conjunction at the nickel electrode. However the changes in impedance that occur as the cell is reversed do provide an excellent qualitative indication of the relative magnitude of internal short circuits, Fig. 4(a). As a short circuit forms and grows the impedance decreases, with the impedance at lower frequencies decreasing most markedly. Eventually if the short-circuit resistance R_{sh} becomes much less than either of the faradaic impedances in Fig. 5, the faradaic impedances are negligible, and the impedance becomes a simple parallel RC circuit (the electrolyte resistance R_s is typically negligible). Curve C in Fig. 4(a) approaches this condition.

The process of recharging a cell that has been reversed requires that short circuits that are formed during reversal be broken before recharge can begin. Figure 3 suggests that some time is required for a short breaking process to occur upon recharge. The impedance of a shorted NiCd cell during a 1 A recharge is shown in Fig. 6 at various points prior to the short breaking. As the recharge proceeds the impedance curve is clearly forming the overall shape that it had in Fig. 4 prior to shorting. The short circuit broke completely about 0.5 hr after the last impedance measurement in Fig. 6, the cell voltage rose to 1.36 V, and the cell started normal recharging.

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RESISTIVE COMPONENT (Ω)
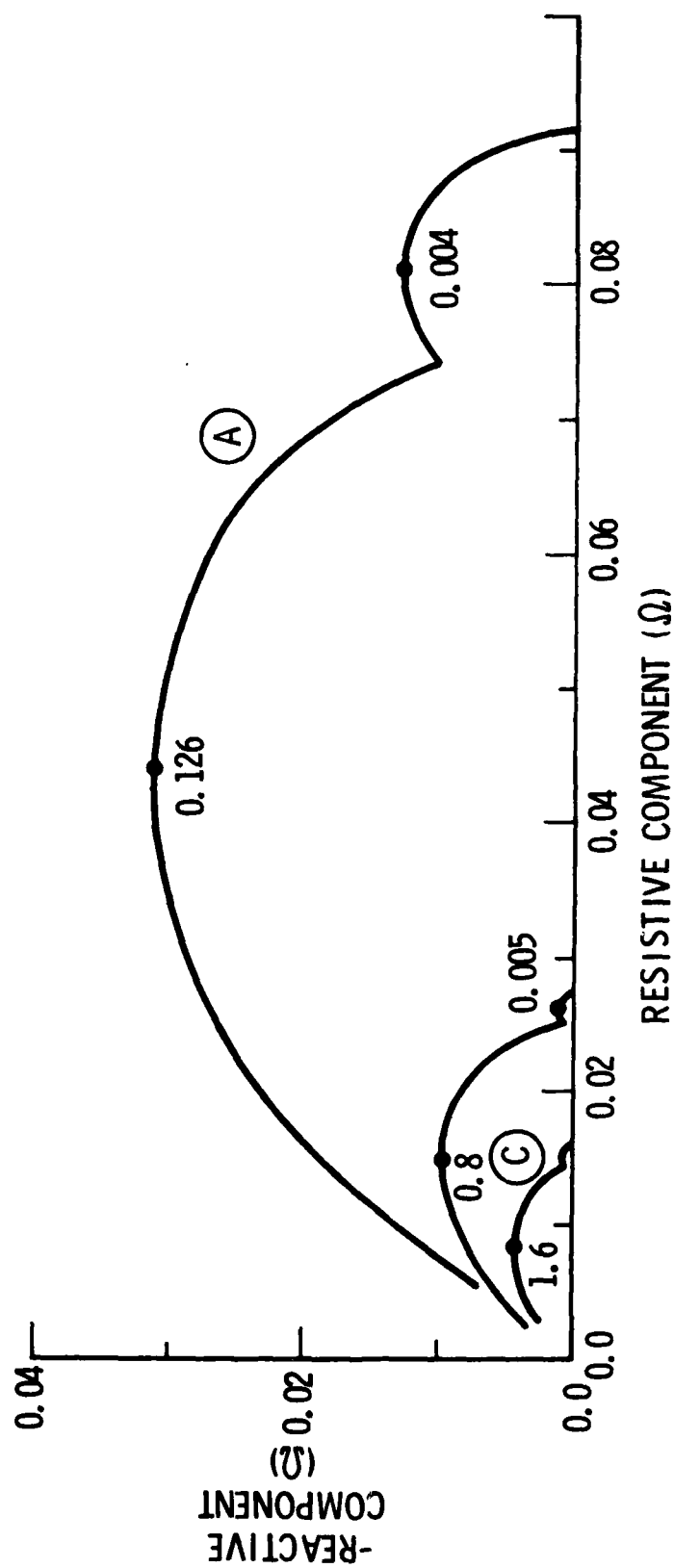
-REACTIVE COMPONENT (Ω)

Fig. 6. Impedance of NiCd Cell During Recharge at 1 A (C/10). Curve C is the initial impedance, curve B after 2 hr, and curve A after 3 hr. After 3.5 hr, the cell voltage rose abruptly to 1.36 V, and it began accepting charge. The numbers indicate frequency in Hz.

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VI. EFFECTS OF CELL REVERSAL ON PERFORMANCE

The changes that can occur in a NiCd cell as a result of repetitive reversals are of interest because this treatment may degrade battery perfor-mance over the long term. No significant degradation in cell performance has been observed during the 2-year study with the cells reversed and shorted up to about 100 times. The most significant performance changes were during cell reversal. As the cells were subjected to reversals it became increasingly easier to form internal short circuits. During the first few reversals, short circuits were not formed at C/100 rates or higher because excessive hydrogen pressurization limited the duration of reversal. But after more than 50 reversals it became difficult to generate appreciable hydrogen at a C/10 rate because shorting occurred so quickly. This behavior suggests that the shorting structure of cadmium metal that is formed in the separator area during reversal is not completely reoxidized upon recharge. Although this residual cadmium metal in the separator area did not appear to affect perfor-mance, it is possible that the susceptibility of the cell to hard-short forma-tion during high rate overcharge is increased. In any case it is clear that conditions of high rate overcharge are to be avoided during cell operation.

The amount of hydrogen evolved during reversal and the time required for a short circuit to form were highly dependent on the state of discharge of the nickel electrode. If the cell was reversed at C/10 immediately after a capa-city discharge, short-circuit formation took place within minutes with essen-tially no hydrogen evolution. However if all residual capacity was discharged from the nickel electrode after the capacity discharge by short circuiting the cell overnight, subsequent reversal at the C/10 rate could lead to extensive hydrogen evolution before internal short circuiting. This result is most likely caused by the active material at the nickel electrode becoming electri-cally nonconductive in the discharged state. Under these conditions the cad-mium dendrites must grow from the nickel electrode current collector to form a short circuit by contact with the cadmium electrode. When the active material at the nickel electrode possesses some residual capacity, it is electrically conductive and is likely to support low rate cadmium reduction in reversal.

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One other significant performance change was observed briefly during several cell reversals. When the cell was reversed, instead of evolving hydrogen at a cell potential between −0.2 and −0.3 V or short circuiting, the cell voltage dropped to −1.4 V, and it began to evolve both hydrogen and oxygen. After several recharge-discharge-reversal cycles, this behavior disappeared as suddenly as it appeared. This kind of behavior is likely to be the result of passivation or depletion of the cadmium electrode. This phenomenon could not be reproduced, and it is not clear as to the conditions that initiated it.

The other potential problem with cell performance was observed with a laboratory constructed cell, under conditions that would not normally occur in sealed NiCd cells. A laboratory cell was flooded with electrolyte and briefly reversed at the C/10 rate. Hydrogen evolution occurred almost exclusively (this was the first reversal for this cell). After recharge, the cell exhibited approximately 20% of its capacity (C/50 rate) as a second discharge plateau at about 0.8 to 0.9 V. Prior to reversal only 5 to 10% of the capacity was discharged at this lower plateau. Upon cell dissassembly severe blistering of the nickel electrode had occurred. This was apparently caused by hydrogen evolution in the compressed plate pack in an environment where all the pores of the sintered electrodes were flooded with electrolyte. Under these conditions it is likely that gas transport out of the sintered nickel electrode was so slow that the bubble pressure in the sinter structure increased to the point where blistering took place. Such blistering is likely to cause isolation of active material from efficient charge and discharge, thereby causing discharge of some capacity at significantly lower potentials than normal.

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VII. CONCLUSIONS

Reduction of $Cd(OH)_2$ to cadmium metal at the nickel electrode can effectively compete with hydrogen evolution during the low rate reversal of NiCd cells. If this reduction reaction occurs for a long period of time, the cadmium metal will form an internal short circuit. After the short circuit becomes relatively solid, it is possible to reverse the NiCd cell at high rates with no hydrogen evolution. No evidence has been found that such short circuiting permanently affects the charge discharge characteristics of the cell. When a cell that has been shorted by reversal is recharged, the cadmium metal short circuit is initially oxidized, after which the cell accepts charge normally.

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